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Explorative Study of NMR Drilling Fluids Measurement

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ABSTRACT

Advanced measurements on drilling fluid samples using Nuclear Magnetic Resonance (NMR) relaxation time measurements at 2 MHz proton resonance frequency were performed. These were carried out on a water-in-oil emulsion (*invert emulsion*) and on an oil-based drilling mud system with varying oil:water (O:W) content.

Longitudinal relaxation time T1 and transverse T2 relaxation time were measured. Good correlation between mean T1 and T2 and varying O:W contents was observed both for emulsions and whole mud.

In a separate measurement series, static barite sagging behaviour of an oil-based drilling mud was observed using a 1D profile experiment in a gradient field.

INTRODUCTION

Drilling fluids are essential for drilling operations. They are designed to serve many different purposes and functions as for example cuttings removal, control of formation pressures and maintaining wellbore stability, to name a few. These fluids are complex slurries whose properties are adjusted by suspending minerals (e.g. barite, clay, calcite) and speciality chemicals into water (water-based fluids) or into water-in-oil emulsions (oil-based fluids). The type, combination and concentration of the components provide fluids with desired properties, as for example density, rheology and filtration properties. It is a requirement for safe and efficient drilling operations to regularly test and monitor these properties, as well as to maintain or re-adjust them.

Test methods for drilling fluids have not changed much over the last 50 years. An acknowledged standard is the recommended practice RP 13 of the American Petroleum Institute (API). For field testing, API RP 13B[1,2] is the relevant standard; while for laboratory testing, API RP 13I[3] is used.

Recent developments regarding remote monitoring and control of drilling operations has also triggered interest in automation of drilling fluid measurements. While one approach to follow-up may be to mechanize and automate standard measurement methods, another option may be to explore alternative methods. Nuclear Magnetic Resonance (NMR) technology is considered as an interesting candidate.

NMR has been used in many applications in the oil and gas industry[4,5,6,7,8,9,10]. Based on the evaluation of previous applications, there may be a strong potential that NMR can also be applied for measuring drilling fluid properties. NMR-measurements on drilling fluids have at least two strong advantages. They are:

- Non-invasive (measurement mechanism is not in contact with the fluid), and
- More versatile (one NMR-device can perform several types of measurements)

EXPERIMENTS

For experiments, two measurement series were run on either water-in-oil emulsion (*invert emulsion*) or a complete oil-based mud (OBM) system. Both fluids were prepared with varying O:W ratio.

Experiments were divided into two main parts, which were:

1. Standard (API) laboratory measurements.
2. NMR relaxation measurements.

Emulsion System

The base-oil that was used to prepare the emulsion was EDC 95/11 *(Total Raffinage Distributions, France)*.

The O:W ratios that were used in the emulsion systems were 70/30, 75/25, 80/20, and 85/15, respectively.

The base-oil has a viscosity of 2.65 cp at 40°C. The viscosity at 35°C was calculated by extrapolation to 2.96 cp. A temperature of 35° C is the operating temperature of the magnet in the NMR benchtop instrument.

To create stable emulsions, Versavert PE and Versavert SE (both products of *M-I SWACO, USA*) were used as the emulsifier.

Oil-based Mud System

An oil-based mud system, with a density of 1.15 s.g., was prepared using EDC 95/11 as the base-oil. The same emulsifiers as above were used. Versavert F (product of *M-I SWACO, USA*) a resin-type fluid loss reducer and Bentone 38 (product of *M-I SWACO, USA*) an organophilic, surface treated clay were used as additives to adjust viscosity and fluid loss control. As a weighting material, Barite was used.

Laboratory Measurements

Laboratory measurements were performed on OBM samples. Three standard API measurements were carried out to characterize the OBM samples:

1. Rheology measurements
2. Electrical stability measurement
3. HTHP filtration loss testing

The result of those measurements is shown in Table 1, 2, and 3 respectively.

Table 1. Results of rheology measurements.

Sample	PV (cp)	YP (lbs/100ft^2)	AV (cp)
OBM – 85/15	18	2	19
OBM – 80/20	19	6	22
OBM – 75/25	20	8	24
OBM – 85/15	23	12	29
OBM* – 85/15	20	10	25
OBM* – 80/20	23	14	30
OBM* – 75/25	26	18	35
OBM* – 85/15	33	24	45

*samples were aged at 150°C for 16 hours

Table 2. Results of electrical stability measurements.

Sample	ES (volts)
OBM – 85/15	600
OBM – 80/20	560
OBM – 75/25	450
OBM – 85/15	360
OBM* – 85/15	990
OBM* – 80/20	770
OBM* – 75/25	490
OBM* – 85/15	440

*samples were aged at 150°C for 16 hours

Table 3. Results of HTHP filtration loss testing.

Sample	Fluid loss (ml)
OBM* – 85/15	1.8
OBM* – 80/20	1.6
OBM* – 75/25	1.4
OBM* – 85/15	1.2

*samples were aged at 150°C for 16 hours

NMR Measurements

NMR measurements were performed using a MARAN Ultra bench-top NMR spectrometer (*Oxford Instruments)*[11]. This instrument is working with an operating permanent magnet and a proton resonance frequency of 2 MHz.

Emulsion and OBM samples were placed in small sample tubes with a volume of 20 ml. The samples needed to be thermostated to 35° C in an oven before NMR-measurements.

Three different types of measurements were performed on the fluid samples:

1. T2 relaxation using the CPMG pulse sequence.
2. T1 relaxation using the inversion recovery method.
3. 1D Profile experiment

Both T1 and T2 measurements were performed on emulsion and OBM samples.

1D profiles were acquired using the PROFILE pulse sequence. The profile pulse sequence resolves the NMR signal spatially along the length axis of the NMR tube by application of a magnetic field gradient. Measurements were performed only on OBM samples. The samples were aged statically in an oven at a temperature of 65° C for 5 days. After aging, the samples were reconditioned to 35° C before they were introduced into the magnet and measured.

System specifications of the NMR spectrometer are shown in Table 4. Application parameters for the T2-CPMG experiment, the T1 experiment, and the 1D profile experiment are shown in Table 5, 6, and 7 respectively.

Table 4. NMR spectrometer system parameters.

Spectrometer frequency	2.12 Mhz
90° pulse length	15.45 µs
180° pulse length	30.9 µs

Table 5. Application parameters for CPMG experiment.

Pulse sequence	CPMG
Number of scans	24
Relaxation delay	10 s
Number of echoes	5120
90-180° interecho spacing	700 µs

Table 6. Application parameters for T1 experiment.

Pulse sequence	Inversion recovery
Number of scans	12
Relaxation delay	10 s
Data points	24

Table 7. Application parameters for 1D profile experiment.

Number of scans	12
Relaxation delay	10 s
Pre gradient pulse length	100 µs
Gradient pulse length	2000 µs
Pre acquisition settle length	1000 µs
90-180° interecho spacing	4000 µs
Gradient amplitude	300

RESULTS AND ANALYSIS

Emulsion Samples

The result of the CPMG experiments on emulsion samples are shown in Table 8. As the starting point, EDC 95/11 was measured without emulsification of water. This measurement is used as a reference value for measurements of emulsion samples with varying O:W ratio.

The mean T2 results from the CPMG experiment show an increasing value as the oil content of the emulsion is reduced.

Mean T2 values are increasing because water relaxes slower compared to oil[12]. The (single exponential) T2 value of water is about 3000 ms.

Table 8. T2 relaxation data of emulsion samples.

Sample	Mean T2 (ms)
EDC 95/11 – 100/0	504
EDC 95/11 – 85/15	612
EDC 95/11 – 80/20	633
EDC 95/11 – 75/25	747
EDC 95/11 – 70/30	750

Fig. 1 shows that the mean T2 results correlate to variations in O:W content. A Pearson correlation coefficient (r) of 0.969 (with $r=\sqrt{R^2}$) is calculated. For five measurements this coefficient indicates that the correlation is significant within 99 % confidence.

The signal-to-noise relationship of the measurements was constant, but in general poor with S/N between 50 and 55. Further experimentation and optimization of the measurement setup is obviously necessary to approach good quality correlations for all combinations.

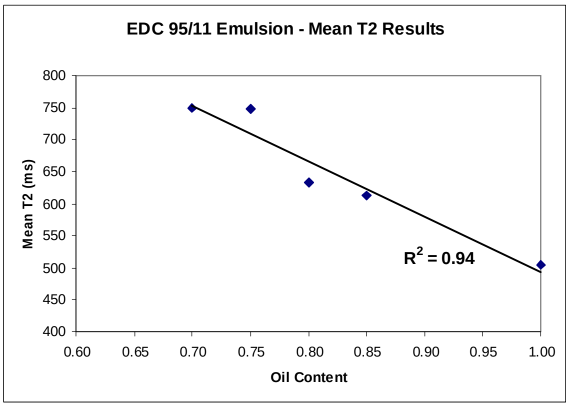

Figure 1. Mean T2 results of EDC 95/11 and water emulsions.

Results of the T1 experiments are similar to the T2-CPMG measurement results. Mean T1 values are increasing as the oil content of the emulsion is decreasing. Mean T1 values are also approximately equal to mean T2 values. This indicates that for the chemicals used, only bulk relaxation processes affect both T1 and T2 relaxation times on emulsion samples and that surface relaxation processes can be neglected[12].

Table 9. T1 relaxation data of emulsion samples.

Sample	Mean T1 (ms)
EDC 95/11 – 100/0	482
EDC 95/11 – 85/15	529
EDC 95/11 – 80/20	630
EDC 95/11 – 75/25	632
EDC 95/11 – 70/30	736

Mean T1-and T2-relaxation times were calculated using a multi exponential fitting routine following with the instrument software package (WinDXP, Oxford Instruments)[11].

OBM Samples

The result of the CPMG experiment on OBM samples is shown in Table 10. Four initial (unaged) mud samples and four aged mud samples were evaluated.

Table 10. T2 relaxation data of OBM samples.

Sample	Mean T2 unaged (ms)	Mean T2 aged (ms)
OBM – 85/15	29	26
OBM – 80/20	33	29
OBM – 75/25	34	33
OBM – 70/30	37	36

Fig. 2 shows that the mean T2 values are increasing as the oil content is decreasing for all OBM samples. This is equivalent to the measurement on the emulsion samples. However measurements on OBM samples give much lower mean T2 values than the emulsions only.

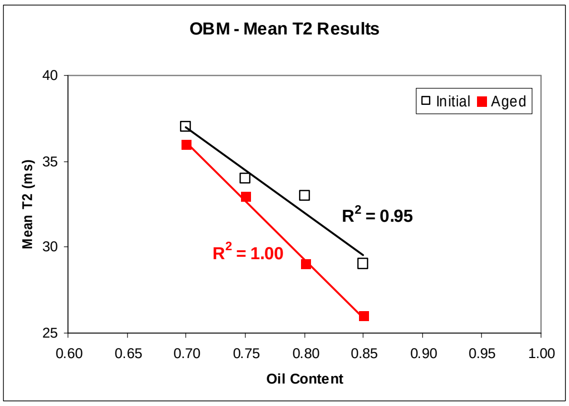

Figure 2. Mean T2 results of OBM sample.

OBM system consists not only of oil and water but also liquids and solid additives in order to provide desired properties to the mud.

The liquid in the neighbourhood of solid particles relax faster due to surface relaxation processes; while liquid at some distance from particles relax affected by bulk relaxation and diffusion (which takes longer time)[5].

Addition of solid additives into the mud has a huge effect, and results in lower T2 values for the mud samples compared to the solid-free emulsions.

Fig. 2 shows that measurements of T2 relaxation data give highly significant correlations between O:W content and mean T2 value.

Standard measurements[2] of O:W content are often quite inaccurate and also take a lot of time in the laboratory. One measurement of T2 relaxation data, using the specified NMR parameters, took approximately five minutes to finish. With proper calibration, it may be possible to measure O:W content of oil-based mud faster and with higher accuracy using NMR technology.

The results furthermore document the importance of ageing drilling mud samples for laboratory measurements. The reduction in T2 values of the aged samples compared to unaged, may be related to improved homogeneity of the sample and full dispersion of all solids components.

1D Profile Experiment

A gradient amplifier to produce a gradient field is needed to perform 1D profile experiments. In this study, measurements were performed using a MARAN Ultra bench-top NMR spectrometer (*Oxford Instruments)*[11] which is equipped with a gradient amplifier, slightly different to the one that was used for the relaxation time measurements. This instrument is also working with an operating permanent magnet and a proton resonance frequency of 2 MHz.

1D profile experiments were performed on four OBM samples with varying O:W ratio that were statically aged inside an oven at a temperature of 65° C for 5 days. The purpose of static aging was to observe settling of solids (barite sagging) at the bottom of the sample tube.

The OBM samples placed in the sample tubes create a fluid column with a height of 6 cm. In the graphic representation of the results (see Fig. 3), 0 cm refers to the bottom of the fluid column and 6 cm refers to the top. The relative NMR-signal can be interpreted as a relaxation time weighted signal amplitude. High relative NMR signal values in the profile reflect the signal of largely solid-free, pure liquid (oil); and low relative NMR signal values reflects the signal from a liquid phase strongly affected by some solids accumulations.

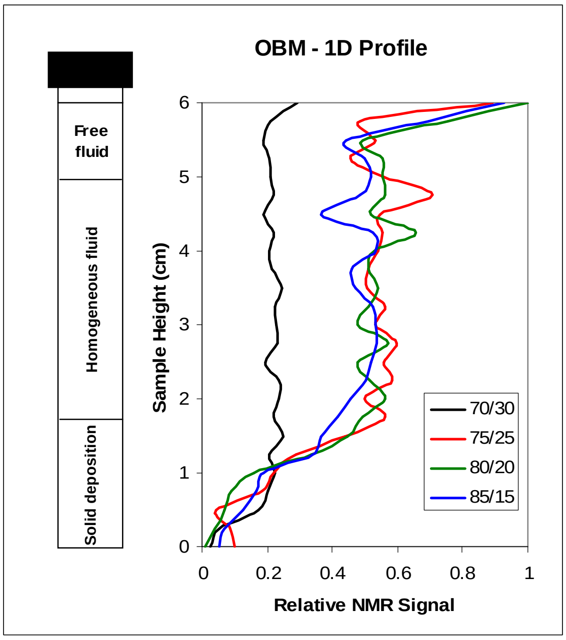

Figure 3. 1D profile of OBM sample.

The sample with an O:W ratio of 70/30 shows a relatively homogeneous mud suspension, as seen in Fig. 3 by the black curve. NMR signals acquired for this sample show very little fluctuation from the top of the sample to about 1 cm above the bottom. Then the relative NMR-signal drops quickly to close-to-zero values at the bottom of the sample. This can be interpreted as a 1-cm accumulation of solids at the bottom of the sample.

A more fluctuating relative NMR signal is observed in samples with 75/25 and higher O:W ratio, see Fig. 3. A high signal approximately 0.5 cm at the top of the sample shows pure oil, most probably due to syneresis. At a sample height from 5 cm to 1.5 cm the signal fluctuates between 0.4 to 0.6. Then the signal starts to decrease strongly at the bottom of the sample where again accumulations of solid materials may have formed.

The highest amount of accumulated solid materials at the bottom of the sample is seen for the sample with an O:W ratio of 85/15. The relative NMR signal shows that accumulations of solids may have a thickness of 2.5 cm.

There is an interesting fact that accumulations of solids increase as the oil content is increasing. To provide constant density, the amount of barite is increased as the oil content is increased. With more barite, accumulations of barite at the bottom of the sample appear to increase.

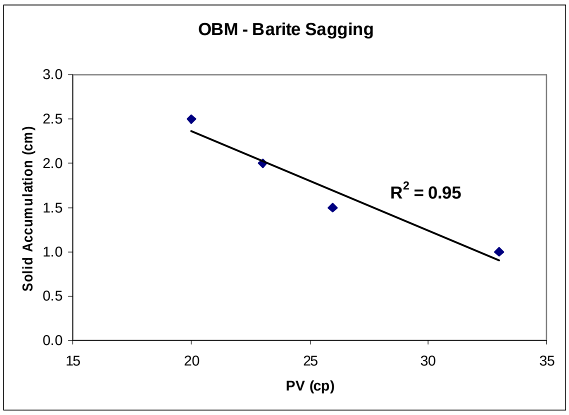

Figure 4. Solid accumulation vs. plastic viscosity of OBM sample.

Fig. 4 shows a correlation between Plastic Viscosity, PV, and solid accumulations at the bottom of the sample. Solid accumulations are decreasing as the viscosity of the mud is increasing. Solids accumulation values were acquired from the interpretation of Fig. 3, where the relative NMR signals start to decrease strongly at the bottom of the sample.

Based on the Stokes' Law[13]; the higher the viscosity, the slower the settling velocity of solid particles. That may explain the trend shown in Fig. 4. It shows a very good correlation and significant within 99 % confidence limits.

From the discussion above, it is concluded that 1D profile experiments may be able to successfully describe the static barite sagging behavior in OBM samples. This could be applied as a new method of barite sagging measurements in the laboratory.

CONCLUSIONS

After performing the experiments and analyzing the results, several conclusions can be drawn:

1. NMR measurement results, e.g. T1 and T2 relaxation data, show very good correlation with O:W content of the prepared emulsions and OBM samples. Mean relaxation time is increasing as the water content of the sample is increasing.
2. NMR relaxation time measurements may be applied as a new method to measure O:W content of mud samples in the laboratory. Our experience is that the measurements require substantially less time than conventional measurements (e.g. retort test).
3. 1D profile experiments using a gradient field have been tested to describe static barite sagging in OBM samples. 1D profiles may allow to characterize inhomogeneities in OBM samples that may correlate to barite accumulation at the bottom of small sample tubes and an oil-phase, most probably due to syneresis, at the top of an OBM sample.
4. There is a correlation between mud viscosity and barite accumulations at the bottom of the sample tubes, indicating that samples with the highest viscosity cause least accumulation.

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